ANALYSIS OF MERCURY IN WATER BY THERMAL DECOMPOSITION, AMALGAMATION, AND ATOMIC ABSORPTION SPECTROPHOTOMETRY

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1. SCOPE AND APPLICATION

This standard operating procedure (SOP) details the analysis of mercury in water samples by thermal decomposition, amalgamation, and atomic absorption spectrophotometry (TDA/AAS). The calibration range is 0.09 ng – 10 ng (0.3 µg/L – 30 µg/L). The method detection limit (MDL) is 0.03 ng (0.1 µg/L). The practical quantitation limit (PQL) is 0.09 ng (0.3 µg/L). Laboratory tests covered by this SOP are W-HG-TDA-R, W-HG-TDARF, and TCLP-TDA.

2. SUMMARY OF THE METHOD

2.1 This SOP is based on EPA method 7473, Revision 0.

2.2 Mercury is liberated from aqueous samples in the NIPPON MA-3000 instrument by controlled heating in an oxygenated decomposition furnace. The sample is pipetted into a ceramic boat and placed on one of the autosampler's stainless steel racks, where 10 ceramic boats fit on each rack, for a total of 100 positions. The sample is dried and then thermally and chemically decomposed within the decomposition furnace. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace. Oxidation is completed and halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to a gold amalgamator that selectively traps mercury. The system is flushed with flowing oxygen to remove the remaining gases and

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decomposition products, followed by the rapid heating of the gold amalgamator to release the mercury vapor. The mercury vapor is then carried by flowing oxygen through absorption cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance is measured at 253.7 nm as a function of mercury concentration.

2.3 The dual-cell NIPPON MA-3000 contains two absorption cells with different path lengths for measuring varying concentrations of mercury in the sample. The NIPPON software automatically chooses which peak to use for sample measurement based on the absorbance. Cell 1 (LOW) is used for concentrations up to 10 ng. Cell 2 (HIGH) is used for concentrations greater than 10 ng up to 600 ng.

Note: Cell 2 is not used during the analysis of water samples. Samples with a concentration greater than 10 ng will be diluted to bring the concentration into the range of Cell 1.

2.4 Co-absorbing gases, which are common interferences, such as free chlorine and certain organics, are purged from the analytical system after release by the decomposition furnace and the mercury vapor is selectively trapped on the gold amalgamator.

2.5 Memory effects: Mercury vapor may remain in the combustion tube, gold amalgamator, or absorbance cells and be released in a subsequent analysis resulting in a positive bias. For example, this may occur when a low concentration sample is analyzed after a sample of high mercury content. To prevent memory effects, the NIPPON MA-3000 automatically performs a blank check after samples containing more than 5 ng of mercury in the water matrix and will repeatedly run a purge cycle until the auto-blank value is below 0.01 ng.

3. APPARATUS AND EQUIPMENT

3.1 NIPPON MA-3000-1, Serial # 2174-0758.

3.2 NIPPON MA-3000-2, Serial # 2174-0759.

3.3 Ceramic boats, part number 440531.

3.4 Eppendorf pipette: 100 – 1000 µL adjustable volume.

3.5 Eppendorf pipette: 1 – 10 mL adjustable volume.

3.6 Fisher brand pipette: 1 – 10 µL adjustable volume.

3.7 Class A volumetric flasks – various volumes.

3.8 Disposable beakers.

4. REAGENTS AND CHEMICALS

See DEP SOP MT-069 for standard and reagent documentation, labeling requirements, and expiration dates. All bottles of reagents and standards (purchased and prepared) in

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the laboratory are required to have Laboratory Information Management System (LIMS) labels, which include a serial number.

4.1 Catalyst – The catalyst is a proprietary patented (JP 5001419) consumable reagent designed and produced by Nippon Instruments Corporation (NIC) that is pre-filled in the instrument's sample heating/combustion tube. It does not have an expiration date. After 2000 measurements, the NIPPON MA-3000 software will display a message to install a new sample heating tube, pre-filled with catalyst. This is a general recommendation as performance may be degraded, but the lifetime of the catalyst depends on the type of samples analyzed and will vary for each catalyst. See the NIPPON MA-3000 Instruction Manual for the sample heating tube replacement procedure.

When a new sample heating tube with catalyst is installed, log the catalyst into the LIMS Standard Preparation Tracker to generate a unique ID. Enter the identity, manufacturer, and lot number. Print the label which contains the unique ID for the catalyst and place it in the maintenance logbook alongside the installation date. Be sure to reset the measurement counter in the instrument software.

The conditioning procedure for the catalyst, if it is new or has not been used for a long time, is as follows:

4.1.1 Analyze 100 mg of flour using a max start temperature of 200 °C, a one-minute drying time at a temperature of 200 °C, and a purge time of 60 seconds.

4.1.2 Analyze 0.1 mL of 5 % HNO_3 using a max start temperature of 200 °C, a 2-minute drying time at a temperature of 650 °C, and a purge time of 60 seconds.

4.2 Carrier gas delivered by cylinder – Oxygen, purity ≥ 90%, pressure 0.2 - 0.5 MPa, regulated to 0.05 - 0.07 MPa.

4.3 Deionized (DI) water – Provided on tap at the laboratory sink and stored in clean wash bottles.

4.4 L-Cysteine in 0.2% HNO_3 – Weigh 100 mg of L-Cysteine and dissolve in 1 L volumetric flask with DI water. Add 2 mL of HNO_3. Top off with DI water to 1 L. Mix well.

4.5 Standards – Preparation of all Hg standards must be documented in the LIMS Standard Preparation Tracker, where a label shall be printed and attached to the bottle or flask that identifies the standard with the name, preparation date, expiration date, and serial number. See DEP SOP MT-069 for standard documentation requirements and expiration dates.

4.5.1 Primary Stock Standard, 1000 mg/L - Commercially available inorganic mercury stock standard from SPEX, AccuStandard, Baker, Baxter, Fisher, VSR, etc. The expiration date is assigned by the manufacturer, and the standard is stored at room temperature. This primary stock standard is used to prepare the calibration standards.

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4.5.2 WTDA-1, 10 mg/L - Pipette 1.0 mL of the 1000 mg/L purchased primary standard into a 100 mL volumetric flask containing 50 mL of L-Cysteine solution in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.3 WTDA-2, 1.0 mg/L - Pipette 0.1 mL of the 1000 mg/L purchased primary standard into a 100 mL volumetric flask containing 50 mL of L-Cysteine solution in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.4 WTDA-3, 100 µg/L - Pipette 1.0 mL of WDTA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.5 WTDA-6, 30 µg/L - Pipette 0.3 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.6 WTDA-8, 20 µg/L - Pipette 0.2 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.7 WTDA-9, 15 µg/L - Pipette 0.15 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.8 WTDA-12, 3.0 µg/L - Pipette 0.3 mL of WTDA-2 (1.0 mg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.9 WTDA-13, 1.0 µg/L - Pipette 0.1 mL of WTDA-2 (1.0 mg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

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4.5.10 WTDA-15, 0.3 µg/L - Pipette 0.3 mL of WTDA-3 (100 µg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.11 Calibration Standards - The calibration standards are prepared from standards WTDA-6 – WTDA-15. Upon generation of a calibration curve, pipette 300 µL of each required WTDA standard into a unique ceramic boat prior to analysis. The concentration for each calibration standard is shown below.

4.5.11.1 Cell 1 (LOW) Calibration Standards:

WSTD2 (0.09 ng / 0.3 µg/L) [300 µL of WTDA-15]

WSTD4 (0.3 ng / 1.0 µg/L) [300 µL of WTDA-13]

WSTD5 (0.9 ng / 3.0 µg/L) [300 µL of WTDA-12]

WSTD8 (4.5 ng / 15 µg/L) [300 µL of WTDA-9]

WSTD9 (6.0 ng / 20 µg/L) [300 µL of WTDA-8]

WSTD11 (9.0 ng / 30 µg/L) [300 µL of WTDA-6]

Note: WSTD2 serves as W-PQL, WSTD5 serves as WLFB, and WSTD8 serves as WSTD8CCV. See Section 7 for quality control (QC) information and Appendix A for an example sample sequence.

4.5.12 Second Source Stock Standard, 1000 mg/L - Commercially available inorganic mercury stock standard from SPEX, AccuStandard, Baker, Baxter, Fisher, VSR, etc. The expiration date is assigned by the manufacturer, and the standard is stored at room temperature. This purchased standard must be purchased from a different vendor than the primary stock standard used to prepare the calibration standards.

4.5.13 WTDA-1C, 1.0 mg/L - Pipette 0.1 mL of the 1000 mg/L purchased second source standard into a 100 mL volumetric flask containing 50 mL of L-Cysteine in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.14 WTDA-3C, 3.0 µg/L - Pipette 0.3 mL of WTDA-1C (1.0 mg/L) into a 100 mL volumetric flask containing 50 mL of L-Cysteine solution in 0.2% HNO_3. Dilute to volume with L-Cysteine solution and mix well. The expiration date for this prepared standard is 180 days from the preparation date, and the standard is stored at room temperature.

4.5.15 WTDA-CA, Second Source Check Standard, 3.0 µg/L - The second source check standard is analyzed for Cell 1 (LOW) with each batch of

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samples in addition to other quality control (QC) checks (see Section 7). Pipette 300 µL of WTDA-3C into a unique ceramic boat, of which the concentration is shown below.

4.5.15.1 WTDA-CA (0.9 ng / 3.0 µg/L) [300 µL of WTDA-3C]

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Water samples are collected in 500 mL or 1000 mL polyethylene bottles with screw caps and preserved by acidifying with nitric acid to a pH below 2. The preservation is done immediately after sample collection, prior to shipment to the laboratory. The pH is then checked upon receipt, and the samples are stored at room temperature. The maximum holding time is 28 days from sample collection to analysis.

6. SAMPLE ANALYSIS PROCEDURE

6.1 Turn on the computer and open the NIPPON MA-3000 software.

6.2 Turn on the main power switch on the left side of the NIPPON MA-3000.

6.3 Open the main cock of the oxygen gas cylinder (0.2 to 0.5 MPa) and adjust the pressure regulator on the rear panel of the NIPPON MA-3000 until the delivery pressure is between 0.05 MPa and 0.07 MPa. Check the tubes for leaks.

6.4 A dialog box will pop up, asking to check the flow path. Click ‘Yes’. This diagnostic will check the flow rate of oxygen, any leaks, and the operation of V1, V2, and V3 valves. Check the NIPPON MA-3000 Instruction Manual for troubleshooting errors. Once completed, the heaters automatically start and the conditioning process begins.

6.5 Conditioning:

The temperature of each heater is increased with oxygen so that the system is purged of moisture and other impurities that may be adhered to the combustion tube to prevent obstructions and/or breakages. Each heater is heated up to the temperature set for measurement. The set temperature of heater H1 is 150 °C and that of heater H3 is 500 °C. This warmup time will take about 15 to 20 minutes.

6.6 Discharge any liquid that has accumulated in the drain tank into an appropriate container for disposal by turning the roller of the roller clamp downward.

6.7 Calibration:

A new calibration is necessary when any significant instrument maintenance is performed and when new calibration standards are prepared, at least once every six months. For example, a new calibration should be performed after the sample heating/combustion tube (catalyst) or gold amalgamator is replaced.

6.7.1 Navigate to the STD table. Clear the old calibration (do not delete) using the blank page icon and save the new one in the calibration folder. Click

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the ‘STD’ box to open the ‘STD INPUT’ window. Enter the concentrations and volumes of each standard.

6.7.2 Pipette 300 µL of each calibration standard into unique ceramic boats according to Section 4.5.11.1 and place in the autosampler stainless steel rack.

6.7.3 Make sure the ‘Measurement’ box has a check mark next to each standard to be analyzed. Select ‘Run’, then ‘Measurement start’ from the menu or click the ‘Start’ button to begin analysis.

6.7.4 In the pop-up window, navigate to the Documents folder where the data will be stored as an MA3 file type. Name the file in the format ‘*YYYY-MM-DD* WCAL-N2’ and click ‘Save’. The measurements will begin and a pop-up window will appear upon completion. Click ‘OK’.

6.7.5 Navigate to ‘Calibration’ and choose ‘fit calibration in every cell’. The calibration curve is linear. The correlation coefficient of the calibration curve must be ≥ 0.995. The % Relative Error in the calibration is measured at every calibration point. For WSTD2 (0.09 ng, 0.3 µg/L), the % Relative Error must be lower than 30 %. For every other STD it must be lower than 20 %. The software also calculates the deviation (DEV) in % for every point in the calibration.

6.7.6 Save the results. Navigate to ‘File’ > ‘Save As Text’ and save the file with the same name as a CSV file type in the Documents folder. Also save the CSV file to the network at: \\floridadep\data\Labs\Lab3\Chem_Data\Inorganic\Mercury\NIPPON MA-3000-2 in the appropriate folder for the year and quarter.

6.7.7. Fill out the Mercury Calibration Standard Preparation Log MT-HGCAL-STD-1.2 excel form and save the file in the format in the format ‘*YYYY-MM-DD* W1-N2 MT-HGCAL-STD-1.2’ to the network at: \\floridadep\data\Labs\Lab5\Chemistry\InorganicPrep\Mercury\W-HG-TDA-R in the appropriate folder for the year.

6.8 Sample Analysis:

6.8.1 Under ‘Sample’, create a new file for the analysis.

6.8.2 Select the appropriate calibration in the STD table.

6.8.3 Select the Water-R method.

6.8.4 The sequence of samples and quality controls to be analyzed are typed into the sample file. See Appendix A for an example sample sequence.

6.8.5 Daily Calibration Check - Analyze the following calibration standards at the beginning of every analysis. The results must be within 10 % of their expected value.

6.8.5.1 Cell 1 (LOW) Daily Calibration Check:

WSTD4 (0.3 ng / 1.0 µg/L) [300 µL of WTDA-13]

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WSTD9 (6.0 ng / 20 µg/L) [300 µL of WTDA-8]

6.8.6 Second Source Daily Calibration Check - Analyze the second source check standard at the beginning of every analysis. The result must be within 10 % of its expected value.

6.8.6.1 Cell 1 (LOW) Second Source Daily Calibration Check:

WTDA-CA (0.9 ng / 3.0 µg/L) [300 µL of WTDA-3C]

6.8.7 Invert each water sample several times to thoroughly mix. For each sample, pour approximately 0.5 mL of sample into a disposable beaker, then pipette 300 µL of the sample from the beaker to a ceramic boat. Then pipette 5 µL of L-Cysteine solution into the ceramic boat and place the boat in the autosampler stainless steel rack.

6.8.8 Make sure the 'Measurement' box has a check mark next to each quality control and sample to be analyzed in the sequence. Select 'Run', then 'Measurement start' from the menu or click the 'Start' button to begin analysis.

6.8.9 In the pop-up window, navigate to the Documents folder where the data will be stored as an MA3 file type. Name the file in the format '*YYYY-MM-DD* W1-N2' and click 'Save'. The measurements will begin and a pop-up window will appear upon completion. Click 'OK'.

6.8.10 Save the results. Navigate to 'File' > 'Save As Text' and save the file with the same name as a CSV file type in the Documents folder. Also save the CSV file to the network at: \\floridadep\data\Labs\Lab3\Chem_Data\Inorganic\Mercury\NIPPON MA-3000-2 in the appropriate folder for the year and quarter.

6.8.11 Fill out the Mercury Analysis Standard Preparation Log MT-HGANA-STD-1.0 excel form and save the file in the format '*YYYY-MM-DD* W1-N2 MT-HGANA-STD-1.0' to the network at: \\floridadep\data\Labs\Lab5\Chemistry\InorganicPrep\Mercury\W-HG-TDA-R in the appropriate folder for the year.

7. QUALITY CONTROL

7.1 Method Blank, MBLK. One method blank is required per batch of 20 samples. Prepare a solution of L-Cysteine in 0.2% HNO_3. Pipette 0.3 mL of the L-Cysteine solution into a ceramic boat. The result of the method blank must be below the Method Detection Limit (MDL) of 0.03 ng (0.10 µg/L).

7.2 Practical Quantitation Limit (PQL), W-PQL. One PQL standard of 0.09 ng (0.3 µg/L) is required per batch of 20 samples. The PQL standard is prepared by pipetting 0.3 mL of WTDA-15 into a ceramic boat. The acceptable recovery limit for the PQL is 70 – 130 %.

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7.3 Laboratory Fortified Blank (LFB), WLFB. One LFB of 0.9 ng (3.0 µg/L) is required per batch of 20 samples. Pipette 0.3 mL of WTDA-12 into a ceramic boat. The acceptable recovery limit for the LFB is 80 – 120 %.

7.4 Matrix Spike and Matrix Spike Duplicate. One matrix spike and matrix spike duplicate are required per batch of 20 samples. Prepare a matrix spike by pipetting 5.0 mL of sample and 150 µL of WTDA-3 into a 15 mL centrifuge tube and mix well. Pipette 0.3 mL of solution from the centrifuge tube into a ceramic boat. Repeat with the same sample to create a duplicate matrix spike. The acceptable recovery limit for the matrix spikes is 80 – 120 %. The Relative Percent Difference (RPD) must be below 20 %.

7.5 Continuing Calibration Verification (CCV), WSTD8CCV, 4.5 ng (15 µg/L). The CCV shall bracket every 10 samples in the sequence to continually verify the calibration. Pipette 0.3 mL of WTDA-9 into a ceramic boat. The acceptable recovery limit is 80 – 120 %.

7.6 Continuing Calibration Blank (CCB). The CCB shall bracket every 10 samples along with the CCV to continually verify that no contamination is present and that no drift has occurred. Acceptable CCB results are below the MDL of 0.03 ng (0.10 µg/L).

7.7 Calibration Check Standards, WSTD4, 0.3 ng (1.0 µg/L), and WSTD9, 6.0 ng (20 µg/L). These check standards (see Section 6.8.5) are used to verify the calibration at the beginning of every analytical run. Pipette 0.3 mL of WTDA-13 and 0.3 mL of WTDA-8 into unique ceramic boats. The acceptable recovery limit is 90 – 110 %.

7.8 Second Source Check Standard, WTDA-CA, 0.9 ng (3.0 µg/L). The second source check standard (see Sections 4.5.15 and 6.8.6) is used to verify the calibration at the same concentration as the LFB from a second source. This standard is analyzed at the beginning of every analytical run. Pipette 0.3 mL of WTDA-3C into a ceramic boat. The acceptable recovery limit is 90 – 110 %.

8. DATA ARCHIVAL

8.1 NIPPON MA-3000: After measurements are complete, in addition to the MA3 instrument file, a CSV copy of the data shall be saved on the hard drive of the instrument computer in the Documents folder in the format '*YYYY-MM-DD* W1-N2', where '*YYYY-MM-DD*' reflects the date of the analysis. The first letter and number following the date reflects the matrix of the analyzed samples and the sequence number performed on that day ('W1' in this case for the first sequence of water samples; 'W2' for the second sequence, and so on). The second letter and number after the date represents the instrument number ('N1' for NIPPON MA-3000-1; 'N2' for NIPPON MA-3000-2). An additional copy of the CSV file shall be saved to the computer network at: \\floridadep\data\Labs\Lab3\Chem_Data\Inorganic\Mercury in the appropriate folder for the instrument, year, and quarter.

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9. MAINTENANCE

9.1 During routine maintenance of the NIPPON MA-3000, both the instrument and computer shall be powered down and disconnected from the power supply. Close the main cock of the oxygen gas cylinder. Allow the instrument to cool to room temperature prior to performing maintenance.

9.2 Refer to the NIPPON MA-3000 Instruction Manual for replacement of the sample heating/combustion tube (catalyst) and the gold amalgamator.

10. SAFETY/HAZARDOUS WASTE MANAGEMENT

10.1 Always wear a lab coat, nitrile gloves, and safety glasses or protective eyewear while working in the laboratory. Concentrated acids shall be handled in a fume hood.

10.2 Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Extreme care must be exercised in the handling of concentrated mercury standards.

10.3 Samples with a high organic content (e.g. oil-saturated soil) should be analyzed with caution. The sample size should be scaled down, as too large of a sample size may result in the ignition of the sample within the combustion tube.

10.4 Mercury wastes that are generated within the laboratory are environmental pollutants. Their disposal is regulated by the rules that govern industrial dischargers. For the City of Tallahassee, the concentration limit for the disposal of mercury into the city's sewer system is 38 µg/L. ANY waste above 38 µg/L in mercury must be handled as hazardous waste. Refer to DEP SOP HG-013 for proper mercury waste disposal.

11. REFERENCES

11.1 NIPPON MA-3000 Instruction Manual.

11.2 EPA Method 7473, Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation and Atomic Absorption Spectrophotometry.

11.3 DEP SOP LB-027, Standard Operating Procedure for Reporting Qualified Data.

11.4 DEP SOP MT-058, Procedure for Metals Data Review and Authorization.

11.5 DEP SOP MT-069, Metals Standard and Reagent Preparation Procedure.

11.6 Quality Manual for State of Florida Department of Environmental Protection Laboratory, located on the Chemistry Section's website.

11.7 Laboratory Health and Safety Manual, located on the Division of Environmental Assessment and Restoration intranet site.

11.8 DEP-SOP-001/01, FS 1000, General Sampling Procedures.

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11.9 DEP SOP HG-013, Mercury Waste Disposal.

11.10 City of Tallahassee, Discharge Restrictions, Sewers and Sewage Disposal (http://talgov.com/Uploads/Public/Documents/you/learn/library/documents/sewer_use_manual.pdf).

APPENDIX A

Example Sample Sequence

WSTD4
WTDA-CA
WSTD9
Rinse
WSTD8CCV
Rinse
CCB
MBLK (batch 1)
W-PQL
WLFB
Sample
Matrix spike
Matrix spike duplicate
Sample
Sample
Sample
Sample
Sample
WSTD8CCV
Rinse
CCB
etc.

Appendix of Significant Changes

08/30/2019 WSTD1 was removed from the calibration. WSTD2 was changed from 0.06 ng to 0.09 ng. WTDA-15 was changed from 0.2 ug/L to 0.3 ug/L. Requirements for calculating the Relative Error for calibration standards was added in section 7.9.

11/26/2019 Edited section 7.9 to use the RSD of response factors to calculate the Relative Error of the calibration.

12/13/2019 Edited section 7.9 to use the percent recovery of the lowest and mid-point calibration standards as a measure of % Relative Error.

01/25/2023 Re-write of SOP due to the purchase of two new NIPPON MA-3000 instruments and the retirement of the two DMA-80 instruments.

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02/09/2026 Added oxygen carrier gas to Section 4. Updated catalyst information and restructured Section 4. Major additions and elaborations to Section 6. Restructured Section 6. Restructured and elaborated on QC details in Section 7. Moved calibration criteria from Section 7 to Section 6. Updated data archival procedure in Section 8. Referred to DEP SOP HG-013 Mercury Waste Disposal in Section 10 and added as a reference to Section 11. Added reference to City of Tallahassee Discharge Restrictions to Section 11. Updated Example Sample Sequence in Appendix A. Updated the proper name of the NIPPON MA-3000 Instruction Manual throughout the document. Grammatical and formatting changes throughout the entire document. Updated all referenced network file paths.

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